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Fracture Process Zone Studies of High Temperature Structural Ceramics

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13. ABSTRACT *(Maximum 200 words)*
Our work on this AFOSR-funded project successfully completed the PFT database through 1280#161#C for alumina and through 600#161#C for the magnesium aluminate spinel, elucidating the importance of crystal structure and fracture topography on the toughening characteristics. We now have a firm understanding of the relationship between the PFT method and the crack growth resistance curve, and are now developing further insights into the role of the microstructural constituents in the crack growth resistance behavior. A first-generation finite element model of the fracture specimen uses both the moire and PFT data to faithfully predict the bulk behavior. Our examination of the electronic speckle pattern interferometry (ESPI) system for elevated temperature measurements of the crack profile have been discontinued, due to inadequate resolution of the available control software. Therefore, the moire experiments are now being modified for elevated temperature studies. We are initiating a second-generation model of the pullout micromechanism to predict the stress-displacement relationship based on the crystal structure properties, grain size distribution, and elastic properties. In response to the detailed studies by the PFT technique, modified micromechanisms are now being incorporated into the model, including high compliance bridging ligaments, such as asperity loading, grain rotation and ligament bending.

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FINAL REPORT

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Sept. 1995

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2. *OBJECTIVES:* No change from proposal.

3. *STATUS OF EFFORT*

Consistent with the objectives of this current study, three primary experimental or modeling efforts are in progress. These activities include the acquisition of baseline information regarding toughening, as measured by the R-curves and interferometry evaluations of crack opening profiles, post-fracture tensile (PFT) testing of wake zone ligaments, and process zone modeling. Our work on the previous AFOSR-funded project successfully completed the PFT database through 1280°C for alumina and through 600°C for the magnesium aluminate spinel, elucidating the importance of crystal structure and fracture topography on the toughening characteristics. The moire experiments are now being modified for elevated temperature studies. A finite element model of the fracture specimen uses both the moire and PFT data to faithfully predict the bulk behavior. Our examination of the electronic speckle pattern interferometry (ESPI) system for elevated temperature measurements of the crack profile have been discontinued, due to inadequate resolution of the available control software.

Over the last year, new graduate students have been given responsibilities for both the U of H and the UW projects, although D. Tran continues with his PhD effort on the project at the University of Washington. At U of H, existing equipment has been modified for the high cycle fatigue experiments to be conducted on this project, and development of the high temperature facilities continue.

We are initiating a second-generation model of the pullout micromechanism to predict the stress-displacement relationship based on the crystal structure properties, grain size distribution, and elastic properties. In response to the detailed studies by the PFT technique, modified micromechanisms are now being incorporated into the model, including high compliance bridging ligaments, such as asperity loading, grain rotation and ligament bending.

4. *ACCOMPLISHMENTS / NEW FINDINGS*

The ability to model the mechanical behavior based on microstructural properties defines an important link in the design cycle for both the processing and design engineers. Relationships between the microstructure and mechanical properties such as creep and strength have found their way into routine mechanical design for a variety of structural applications. With a mature understanding of the active toughening mechanisms, the elements of the microstructure will eventually be incorporated into appropriate models of flaw tolerance and will be used to develop optimal ceramic microstructures.

The current project, initiated in March of 1993, has focused on the details of the fracture process zone required for the construction of a constitutive micromechanical model of the microstructural role in crack propagation resistance. Two parallel efforts are involved to accomplish this goal. The microstructural characterization of the fracture process zone incorporates the post fracture tensile (PFT) test, conducted at the University of Houston, and the moiré interferometry studies, conducted at the University of Washington, for the determination of the crack face traction distribution and the macroscopic crack opening profile. These two efforts are united to generate a micromechanical model for predicting the fracture process zone behavior in terms of the fundamental material and microstructural properties. It is the ultimate goal of this effort to establish a material design methodology for the prediction of damage tolerant microstructures.

Air Force Relevance

The future design of energy-efficient jet engines, fracture resistant optical windows or structural members for hypersonic vehicles, or thermal insulation for re-entry vehicles, all depend upon a reliable quantification of the appropriate fracture parameters for the prediction of safe service loads.

The microstructure/properties relationship which we are developing in this present effort will provide relevant engineering payoffs for future Air Force activities, such as the development of advanced structural ceramics for hypersonic or subsonic high performance aircraft and unmanned vehicles. Materials systems anticipated for use on such vehicles will be expected to outperform currently used materials in terms of mechanical properties as well as environmental stability at high temperatures, creep resistance and weight-efficiency. Since many of these applications will eventually consider brittle materials, such as ceramics or ceramic composites, the design effort will clearly require some level of damage tolerance from the material, and a fundamental relationship between the processing, the inherent flaw population and the fracture properties. Therefore, the ability to predict the limits of each candidate material system based upon constituent properties will ultimately be the prime governing component of the design-manufacturing loop.

This current modeling effort seeks to accomplish this goal through the development of a general predictive capability for design of the microstructures of brittle materials. To this end, the damage tolerance is addressed thorough the crack growth resistance curve (R-curve) concept, where an assumed inherent flaw population in the ceramic or ceramic composite will nucleate a critical flaw only when the R-curve limits are exceeded. Therefore, enhanced R-curve behavior in an otherwise brittle material will effectively increase the tolerable flaw size, hopefully to a detectable size scale.

Our accomplishments in each of the topic areas are highlighted in the following subsections.

Fracture Process Zone Characterization: Wake Studies (U of H)

Based upon the work reported earlier, the wake compliance studies have been published in the American Ceramic Society Journal and is referenced in Section 6. Two students presently supported on this grant, Xin Dai and Yi Fang, have co-authored further papers which will be submitted soon. Here, we used our post fracture tensile (PFT) results to approach the fracture problem from new directions.

Xin Dai estimated the PFT wake traction parameters from R-curve results published in the open literature. Comparing these functions with our own PFT wake traction functions, we have calculated R-curve for several test specimen geometries which appear in the literature. The role of the microstructure and the specimen geometry are clearly enunciated in this exercise, which further validates the value of the PFT method in modeling efforts.

Yi Fang considered the interrelationship between the microstructure and the R-curve behavior of the BAS / Si3N4 self-reinforced composite which is under study in our lab. On another project in our lab, we have developed control of the self-reinvorcement phase morphology, so through the micromechanical concepts established from the past PFT work, we have shown the relationship between the Si3N4 morphology and size and the shape of the R-curve. This work explores the microstructural response to different processing conditions and identifies the toughening effect associated with controlled variations in whisker morphology. Also, this paper will consider various popular toughness characterization methods on the values reported for the same microstructure.

Over the past year, Krupal Patel has developed the equipment for insitu loading of specimens for crack growth studies in both the optical and SEM environments. With completion of the necessary control software, she will be starting crack growth experiments by mid-October. She will contribute direct information on the microstructural involvement in both the crack path as well as details of wake bridging ligaments. This work will support the third generation modeling effort with Prof. AS. Kobayashi, U of W.

The equipment she is building will also be used to monitor wake and crack tip behavior in cyclic loading conditions. The piezoelectric actuator may be driven by a signal generator to offer a wide range of stress/strain cycles in the tension-tension regime. This will be important to the modeling project when we will be ready to develop the physical basis for the geometric component of the micromechanic model of the general fatigue problem.

Fracture Process Zone Characterization: Cyclic Loading Results (U of H)

This work has been delayed due to difficulties in the equipment. We have been re-working the controller for the cyclic fatigue tests. I expect this problem to be resolved by mid-fall 97. Additionally, three new graduate students have been learning the PFT technique.

The work completed in this area has substantiated existing data for different microstructures. A characteristic compliance transition persists for each of the three microstructures which we have studied by PFT methods, in the small displacement range. Very preliminary results suggest that the alumina microstructures containing higher grain boundary porosity but about the same grain size distribution (ave. grain size = 18 μm.) show a compliance transition with about the same compliance values, but appearing at lower displacements (about 0.06 to 0.07 μm, rather than 0.09 to 0.010). As before, the transition point increased to higher stresses with continued load cycling, and since the upper load limit is controlled, the hysteresis loop area decreased with each cycle. I emphasize that this data is preliminary, and this effort must be continued before publication of these ideas. Should this relationship endure as I expect, there are interesting implications pertaining to the modeling question. First, the interfacial constants needed for complete description of the grain interface separation event clearly depends upon the details of the microstructure, including the distribution of microporostity between the grain interior and the boundaries. Secondly, the stiffness of the bridging ligaments seems to be unchanged within the anticipated experimental error. This information will help in the development of the geometric aspects of the grain separation model, which seeks to establish the micromechanical link between the microstructure and the crack initiation and growth behavior in ceramics in general.

A second set of new data is taken from a course-grained spinel microstructure. Three significant microstructural features differentiate this material from the alumina studied above. a) Grain size distribution: The grain size ranges from about 15 to 500 μm with an average near 75 μm. Significantly larger than the 18μm of alumina. b) Crystal Structure: Although elastic anisotropy characterizes both crystal systems, the cubic spinel has only one thermoelastic constant. This has strong implications in the residual stress state associated with the test conditions. c) Grain boundary phase: The spinel contains a crystalline grain boundary phase. This influences both the high and low temperature behavior, however, the defined melting point oat 825 °C markedly separates the mechanical behavior from that of glass phase containing ceramics, such as alumina.

Preliminary cyclic PFT data on this material shows a distinctly different response from that of the spinel. Figure 1 shows an initial non-closing hysteresis loop, similar to that of the alumina, but subsequent loops close completely, and fall directly upon each other.

For completeness, I provide the following brief summary of work already published in a referenced article, some of which was reported earlier. This background will help to put the new results in perspective.

The stress-displacement curve for one load-unload cycle using the current setup appears in Fig. 2. For this particular test, the load was applied sinusoidally such that the stress varied from 0.15 MPa to 1.25 MPa over a period of roughly 400 seconds. For low numbers of cycles, the load-unload cycle can be described by three linear regions. The first, starting from 0.15 MPa and extending to 0.98 MPa, describes the initial loading response. At first glance, the displacements in Fig. 5 conflict with those in Fig. 2, but in actuality the behaviors agree quite well. Note that in Fig. 2 we do not have data below roughly 3 MPa and that portion of the curve was approximated by a dashed line extrapolating linearly to the origin. It is interesting to note in Fig. 5 that at the transition point (0.98 MPa, 0.1 μm) the stiffness decreases abruptly to a behavior characteristic of the macroscopic loading stiffness in Fig. 2. Therefore, if the second regime in Figure 5 was extrapolated to 3 MPa, the displacement would be in the 1 μm to 2 μm range.

The unloading stiffness appears to evidence a value similar to the initial loading compliance. If the frictional model presented by Guiu et al. (1992) accurately describes the grain-bridging mechanism then we would expect a transition point during the unload cycle, similar to the one noted during the loading portion of the cycle. When frictional mechanisms prevail, the presence of a lower transition point characterizes an abrupt decrease in stiffness again and the displacements return to zero, closing the loop. Instead, Fig. 5 indicates a permanent residual displacement. For this particular load-unload cycle the specimen experiences an increase of approximately 0.11 μm in total crack-face separation. With repeated load cycling, the critical load and displacement defining the transition point increase slowly, evidencing some sort of strengthening mechanism. Therefore, under controlled load conditions the contribution to total residual displacement decreases with each cycle.

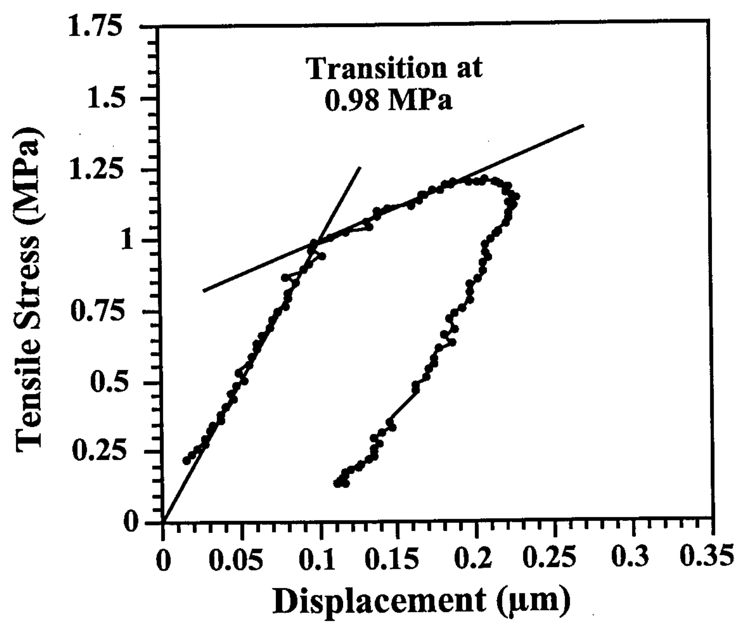

Fig. 2 A typical load-unload cycle for PFT#2.

Fatigue Data (U of H)

The PFT technique offers an opportunity to directly characterize the fatigue behavior of the isolated wake ligaments. Using the closed-loop controller with a piezoelectric actuator, a PFT specimen was fatigued to obtain preliminary information on load cycling at room temperature.

Cyclic Loading of the #2 PFT below critical strain shows no degradation over 10,000 cycles (FIG 1). As reported earlier, All cycles are tension-tension under load control conditions. Increasing the lad to strain beyond critical conditions causes a degradation following extended cycling, implying that the micromechanism "wears out" as the nonrecoverable displacements accumulate. The increase in compliance shown in Fig. 2 implicates a reduction in contact area of the wake faces, or a loss of contact efficiency. Either must result from damage accumulation associated with contact points.

Similar experiments on PFT specimen #1 (cut from the DCB, immediately behind the crack tip) load to the same load level. Since these characteristically stiffer specimens do not exceed the critical displacement, no compliance increase is observed following extended cycling (Fig. 2). Further load increases, however, exceed the transition strain, causing mechanical damage, as above, with the accompanying increase in compliance (Fig. 3).

The PFT technique has been used as a tool for fracture interface degradation studies. We intend to further this effort with increased cycling range to examine the entire domain defined by the PFT stress - displacement curve. Modeling of this phenomenon will include micromechanical parameters to represent the process zone degradation with cyclic loading.

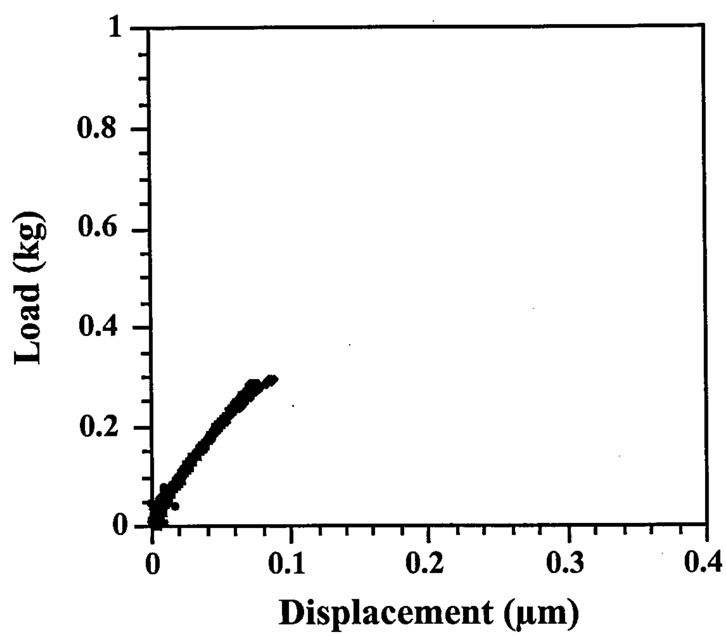

Figure 1. Superposed loading traces of the hysteresis loop for PFT specimen #2. Three compliance traces for one specimen following 3, 100 and 10,000 loading cycles, showing no degradation.

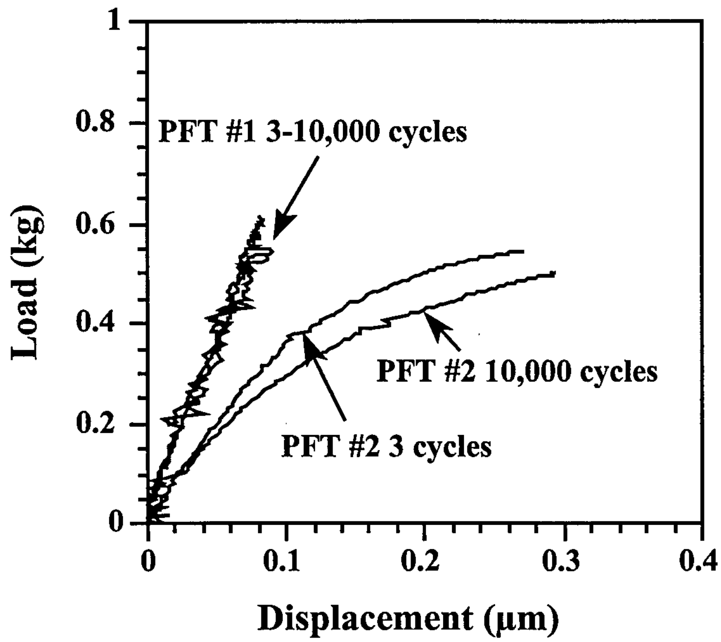

Figure 2. Loading PFT #2 beyond critical displacement of about 0.1 µm shows a degradation in stiffness following 10,000 cycles. The mechanically stiffer PFT #1 loaded to the same load shows no degradation, as strains have not exceeded critical values.

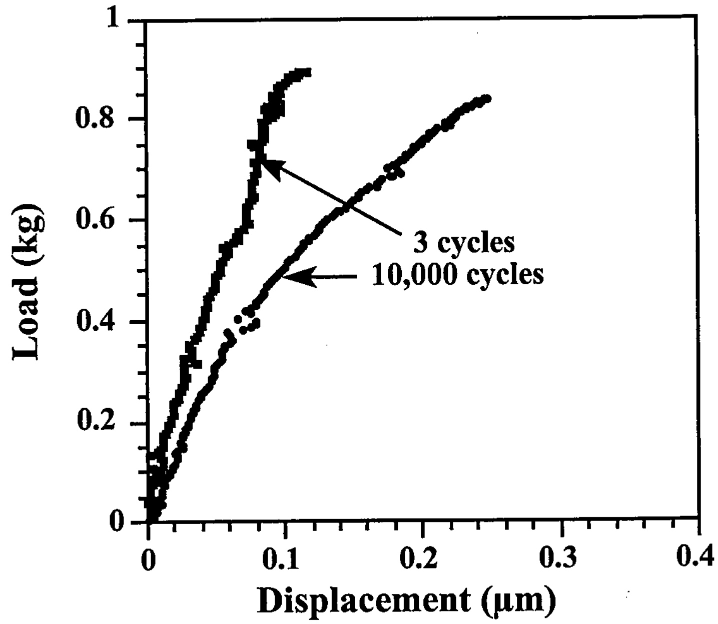

Figure 3. Further cycling of PFT #1 to higher loads will show stiffness decrease when the critical strain is exceeded by increased maximum load.

High Temperature Moiré Interferometry (U of W)

With the acquisition of INNOVA 308 argon ion laser, Coherent, Inc. through a FY95 DURIP grant, we now have the capability of creating a holographic grating of frequency of 1,200 lines per mm on the photoresist film. The process, which was developed during the past year, is to evaporated a thin nichrome film onto the polished surface of an alumina specimen, deposit a photoresist mask of 1,200 lines per mm on top of the nichrome film by interferometry technique and then etch in ceric ammonium nitrate solution. The result is a thin nichrome grating of square dots with a frequency of 1,200 lines per mm on top of the alumina specimen. This grating can be used for Moiré interferometry at testing temperature up to 800°C and possibly to 1000°C which is above the glass transition temperature of the alumina grain boundary phase.

The custom-built furnace from Applied Test System, Inc. was delivered in spring 1994. This furnace with a temperature rating of 1540°C is equipped with quartz windows to measure horizontal displacements on the specimen surface using Moiré interferometry. These windows can be used to measure the same in-plane displacement by electronic speckle pattern interferometry (ESPI). The horizontal displacement measurement restricts the fracture specimen configurations to edge-cracked three point bend and wedge-loaded, double cantilever beam (WL-DCB) specimens.

Phase-Shifting Moiré Interferometry(U of W)

With the above development of a suitable high temperature Moiré interferometry technique, efforts on ESPI has been temporary suspended. The reason for this shift back to Moiré interferometry is due in part to the long lead time for modifying the current software for ESPI use. The acquired software for phase shifting Moiré interferometry

requires considerable modification, more than the vendor had led us to believe, and thus a decision was made to study the high temperature fracture process zone (FPZ) by phase-shifting Moiré interferometry. The necessary hardware, i.e. a CCD camera, a piezoelectric transducer, optical components, a dedicated PC and the software were acquired, assembled and debugged during this report period.

The phase shifting Moiré interferometry was then used to study the FPZ in ceramics at room temperature. The phase shifting provided an added order of magnitude to the sensitivity of 1200 lines/mm Moiré grating which in itself lacked the sensitivity for analyzing the brittle alumina. The utility of the phase shifting Moiré interferometry was demonstrated by a room temperature, slow crack growth study of an alumina WL-DCB specimen. The recorded wedge opening displacement and the crack growth data were used to drive a finite element model of the alumina WL-DCB specimen in its generation mode from which the crack bridging force and the crack growth resistance curve were extracted through an inverse process by matching the measured and recorded crack opening displacements. The dissipated energy in the FPZ was also computed, as expected, were found to be in excess of 99 percent of the energy released during stable crack growth.

Micromechanic Modeling (U of W and U of H)

A first generation, 2-D finite element (FE) model of grain bridging in the FPZ was constructed from a photomicrograph of alumina. Frictional resistance to intergranular pull-out was attributed to the intergranular compressive forces due to the residual thermal stress generated by the anisotropic contraction during the cool down process. The FE model consisted of an assembly of eleven layers of eleven grain sizes without an enforced interlayer compatibility. This FE model was modified by imposing the observed intragranular fracture sites which were generated prior to the post fracture tension (PFT) tests. The friction coefficient for intergranular sliding was also reduced in accordance with experimental data. This model was then used to replicate the post fracture tension (PFT) tests at four locations along the FPZ of a stably grown crack in a high density alumina, DCB specimen. The resultant crack bridging stress versus crack opening displacement relations at three testing temperatures of 20°C, 400°C and 600°C agreed well with the experimentally obtained PFT results. Thus the FE model provided further verification that the residual thermal stresses are the main source of resistance to grain pull out in the FPZ.

FUTURE WORK

High Temperature/Phase-Shifting Moiré Interferometry

Work on high temperature Moiré interferometry with phase shifting will be suspended for three months while the PhD student, Duc K. Tran, prepares for his PhD General Examination. This work is expected to resume in January 1998, starting with trial runs of the test equipment followed by a FPZ study of alumina at 600 - 1000°C.

Micromechanic Modeling

Matthew K. Kokaly, a new PhD student will take over the micromechanic modeling starting September 1, 1997. The first task is to execute the first generation FE of different grain size distributions. The purpose of this numerical experiment is to study the effects of the grain size distributions on the energy dissipated at the FPZ and hence on the fracture toughness. The predicted toughness variations with be compared with

experimental results in published literature. If successful, this study will provide design guideline toward the development of tougher structural ceramics.

The second task is to enforce compatibility between the FE model layers of tension strips, each representing a different grain size category. The effect of grain rotation on grain pull-out as well as a workable criterion for transgranular fracture must also be incorporated in this second generation model.

5. PERSONNEL SUPPORTED:

Faculty: Ken W. White, Associate Prof., University of Houston

Albert S. Kobayashi, Prof., University of Washington

Graduate Students: Yi Fang, University of Houston (New student: June 96)

Xin Dai, University of Houston (New student: June 96)

Richard Geraghty, University of Houston (Part time on this project) (New student: June 97)

Krupal Patel, University of Houston

Duc Tran, University of Washington.

Matt Kokaly, University of Washington

Undergraduate Student: Said AbuShmais, University of Houston

Edwin L.Phillips, University of Houston

6. PUBLICATIONS (Last 12 Months):

Hay, J.C., White, K.W. "Microstructural Interaction in the Wake Cohesive Zone for Small Crack-Opening Displacements," Acta Metallurgica et Materialia, (Sept. 1997).

Hay, J.C., White, K.W. "The Stiffness of Grain Bridging Elements in a Monolithic Alumina," J. Amer. Ceram. Soc., **80**[5] 1293-97 (1997).

Z.K. Guo, A.S. Kobayashi, J. Hay and K.W. White, "Fracture Process Zone of Monolithic Al_2O_3," Int. J. Frac, (1997).

Hay, J.C., White, K.W. "Grain Boundary Phases and Wake Zone Characterization in Monolithic Alumina," *J. Amer. Ceram. Soc.*, **78**[4] 1025-32 (1995).

Z.K. Guo, A.S. Kobayashi and N.M. Hawkins, "Dynamic Mixed Mode Fracture of Concrete," to be published in *Int.J. Solids and Structures*.

C. Ortiz, J.C. Hay, K. W. White, C. Vipulanandan, "Experimental Study on the Strain Softening Behavior of Cement Mortar Using The Post Fracture Tensile Method," Submitted to: Cement and Concrete Research, (1997).

Conference Proceedings and Book Chapters:

"Process Zone Modeling of Polycrystalline Ceramics," D. Tran, A.S. Kobayashi, & K.W. White, in: Advanced Technology in Experimental Mechanics, Wakayama, Japan, July 25-26, 1997.

"A Method for the Determination of Cohesive Zone Tractions," J.C. Hay and K.W. White, Accepted: 9th International Conference On Fracture, eds. R. Ritchie, et al., Pergamon Press, Oxford, 1997.

"Process Zone Modeling of Polycrystalline Alumina," D. Tran, A.S. Kobayashi, J.C. Hay and K.W. White, in: Fracture Mechanics of Ceramics, v.12, eds. R.C.Bradt, et al., Plenum, 1996.

"Wake Process Zone Studies of Elevated Temperature Structural Ceramics,"K.W. White and J.C. Hay, Accepted in: Fracture Mechanics of Ceramics, v.12, eds. R.C.Bradt, et al., Plenum, 1996.

K. W. White, L. Olasz, R. Geraghty, AS Kobayashi,"Interface Degradation in Ceramics under Cyclic Loading," Damage and Failure of Interfaces, Balkema, Rotterdam, Netherlands, 1998.

A.S. Kobayashi, "Dynamic and Impact Failure of Ceramic Composites," *High Temperature Mechanical Behavior of Ceramic Matrix Composites*, eds. S.V. Nair and K. Jakus, Butterworth-Heinemann, pp. 121-52, (1995).

Z.K. Guo, M. Kosai, A.S. Kobayashi and N.M. Hawkins, "Further Studies of the Fracture Process Zone Associated with Mixed Mode Dynamic Fracture of Concrete," to be published in the *Proceedings of Fracture Mechanics for Concrete and Concrete Structures.*

7. INTERACTIONS/TRANSITIONS (Last 12 Months):

a. Meetings, conferences and seminars:

K. W. White "Interface Degradation under Cyclic Loading of Ceramics", Damage and Failure of Interfaces, Vienna, Austria, Sept. 22-24, 1997.

K. W. White "The Role of Microstructures in Cyclic Degradation of Structural Ceramics", University of Ljubljana, Ljubljana, Slovenia, Sept. 26, 1997. Invited

K. W. White "The Role of Microstructures in Reliability of Ceramics", IMM Young Investigators Workshop, GE Research Center, Schenectady, NY, Aug. 20-22, 1997. Invited

K. W. White "Microstructural Scale in Damage Development in Ceramics", Workshop on the Role of Microstructures in Reliability of Ceramics, Center for Reliability of Ceramics, U of H, Houston, TX , Feb. 18-19, 1997. Invited

K. W. White "Cohesive Zone Issues in Monolithic Ceramics", Visiting Seminar, Dept. of Materials Science, University of Utah, May 17, 1996. Invited

K. W. White "Microstructural Response in the Fracture Process Zone of Monolithic Ceramics", Visiting Seminar, Ceramics Division, ORNL, Oak Ridge, TN, May 3, 1996. (Invited)

"Crack Face Bridging in Monolithic Ceramics", J. Hay and K.W. White, 99th Annual Meeting of the American Ceramic Society, Cincinnati, Ohio, May 4-7, 1997.

"Fracture Process Zone Modeling of Monolithic Alumina", D. Tran, A.S. Kobayashi, J. Hay and K.W. White, 99th Ann. Meeting of Amer. Ceramic Society, Cin., Ohio, May 4-7, 1997.

8. NEW DISCOVERIES: None

9. HONORS/AWARDS

K.W. White
Faculty Research Award, Cullen College of Engineering, 1995

A.S. Kobayashi:

Member, National Academy of Engineering, October 1, 1986.

Honoree, International Symposium on Fracture Mechanics and Life Management of Materials and Structures, Tokyo, Japan, July 29, 1994.

M. M. Frocht Award, Society for Experimental Stress Analysis, June 1995.

GE Senior Research Award, American Society of Engineering Education, July 28, 1995